

FORM PTO 1390
(REV 5-95)

US DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY DOCKET NUMBER
2001_1245ATRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. §371U.S. APPLICATION NO.
(of 37 CFR 1.53)
NEW 097936165International Application No.
PCT/NO00/00079International Filing Date
March 6, 2000Priority Date Claimed
March 8, 1999**Title of Invention**

A NEW SILAGE AID, PROCESS FOR PREPARING THIS SILAGE AID AND USE OF THIS SILAGE AID

Applicant(s) For DO/EO/US


Kari AASBØ and Harald BREIVIK

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. §371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. §371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. §371(c)(2)) (in English)
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. §371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. §371(c)(3)).
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19.
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)). (UNEXECUTED)
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. §371(c)(5)).

Items 11. to 14. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☒ Other items or information: (a) Published International Application (WO 00/53033) with attached International Search Report; (b) PCT Request; (c) Forms PCT/IB/301, 304, and 308; (d) International Preliminary Examination Report; and (e) PCT Written Opinion (form PCT/IPEA/408) and response thereto.

U.S. APPLICATION NO. (37 CFR 1.53) NEW 09/936165		INTERNATIONAL APPLICATION NO. PCT/NO00/00079		ATTORNEY'S DOCKET NO. 2001 1245A	
15. [X] The following fees are submitted BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): Neither international preliminary examination fee nor international search fee paid to USPTO and International Search Report not prepared by the EPO or JPO \$1000.00 International Search Report has been prepared by the EPO or JPO \$ 860.00 International preliminary examination fee not paid at USPTO but international search paid to USPTO \$ 710.00 International preliminary examination fee paid to USPTO but claims did not satisfy provisions of PCT Article 33(1)-(4) \$ 690.00 International preliminary examination fee paid at USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$ 100.00 ENTER APPROPRIATE BASIC FEE AMOUNT =				CALCULATIONS	PTO USE ONLY
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$	
Claims	Number Filed	Number Extra	Rate		
Total Claims	30 -20 =	10	X \$18.00	\$180.00	
Independent Claims	4 - 3 =	1	X \$80.00	\$80.00	
Multiple dependent claim(s) (if applicable)			+ \$270.00	\$	
TOTAL OF ABOVE CALCULATIONS =				\$1260.00	
<input type="checkbox"/> Small Entity Status is hereby asserted. Above fees are reduced by 1/2.				\$	
SUBTOTAL =				\$1260.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				+	\$
TOTAL NATIONAL FEE =				\$1260.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40 per property +				\$	
TOTAL FEES ENCLOSED =				\$1260.00	
				Amount to be refunded	\$
				Amount to be charged	\$
a. [X] A check in the amount of \$1260.00 to cover the above fees is enclosed. A duplicate copy of this form is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. 23-0975 in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 23-0975.					
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
19. CORRESPONDENCE ADDRESS			By: <u>Michael R. Davis</u> Michael R. Davis, Registration No. 25,134 WENDEROTH, LIND & PONACK, L.L.P. 2033 "K" Street, N.W., Suite 800 Washington, D.C. 20006-1021 Phone: (202) 721-8200 Fax: (202) 721-8250 September 10, 2001		
 000513 PATENT TRADEMARK OFFICE					

THE COMMISSIONER IS AUTHORIZED
TO CHARGE ANY DEFICIENCY IN THE
FEES FOR THIS PAPER TO DEPOSIT
ACCOUNT NO. 23-0975

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of :
Kari AASBØ et al. : Attn: BOX PCT
Serial No. NEW : Docket No. 2001_1245A
Filed September 10, 2001 :

A NEW SILAGE AID, PROCESS FOR
PREPARING THIS SILAGE AID AND USE
OF THIS SILAGE AID
[Corresponding to PCT/NO00/00079
Filed March 6, 2000]

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents,
Washington, DC 20231

Sir:

Please amend the above-identified application as follows:

IN THE SPECIFICATION

Page 1, after the title of the invention, please insert:

This application is a 371 application of PCT/NO00/00079 filed March 6, 2000.

IN THE CLAIMS

Please amend claims 3-7 and 10-11 as follows:

3. (Amended) Silage aid according to claim 1, wherein the short chain carboxylic acid is formic acid of concentration 60-100%, preferred 83-98%.

4. (Amended) Silage aid according to claim 1, wherein the short chain carboxylic acid is acetic acid or propionic acid of concentration 60-100%, preferred 80-100%.

5. (Amended) Silage aid according to claim 1, wherein 0.1-10% of the antioxidant, preferred 0.3-2%, is dissolved in the short chain carboxylic acid.

6. (Amended) Silage aid according to claim 1, comprising an antioxidant selected from the group BHA, TBHQ and propyl galate (PG); and formic acid.

7. (Amended) Silage aid according to claim 1, comprising BHA and BHT; and formic acid.

10. (Amended) Process according to claim 8, by dissolving 0.1-10% of the antioxidant, preferred 0.3-2%, in formic acid, acetic acid and/or propionic acid of concentration 60-100%.

11. (Amended) Process according to claim 8, for preparation of a silage aid comprising 2,6-di-*tert*-butyl-4-methylphenol (BHT) and 3-*tert*-butyl-4-hydroxyanisole (BHA), and formic acid, by

a) dissolving BHA in the acid, and

b) subsequently, dissolving BHT in the solution obtained in step a).

Please add new claims 14-30 as follows:

14. (New) Silage aid according to claim 2, wherein the short chain carboxylic acid is formic acid of concentration 60-100%, preferred 83-98%.

15. (New) Silage aid according to claim 2, wherein the short chain carboxylic acid is acetic acid or propionic acid of concentration 60-100%, preferred 80-100%.

16. (New) Silage aid according to claim 2, wherein 0.1-10% of the antioxidant, preferred 0.3-2%, is dissolved in the short chain carboxylic acid.

17. (New) Silage aid according to claim 3, wherein 0.1-10% of the antioxidant, preferred 0.3-2%, is dissolved in the short chain carboxylic acid.

18. (New) Silage aid according to claim 4, wherein 0.1-10% of the antioxidant, preferred 0.3-2%, is dissolved in the short chain carboxylic acid.

19. (New) Silage aid according to claim 2, comprising an antioxidant selected from the group BHA, TBHQ and propyl galate (PG); and formic acid.

20. (New) Silage aid according to claim 3, comprising an antioxidant selected from the group BHA, TBHQ and propyl galate (PG); and formic acid.

21. (New) Silage aid according to claim 4, comprising an antioxidant selected from the group BHA, TBHQ and propyl galate (PG); and formic acid.

22. (New) Silage aid according to claim 5, comprising an antioxidant selected from the group BHA, TBHQ and propyl galate (PG); and formic acid.

23. (New) Silage aid according to claim 2, comprising BHA and BHT; and formic acid.

24. (New) Silage aid according to claim 3, comprising BHA and BHT; and formic acid.

25. (New) Silage aid according to claim 4, comprising BHA and BHT; and formic acid.

26. (New) Silage aid according to claim 5, comprising BHA and BHT; and formic acid.

27. (New) Silage aid according to claim 6, comprising BHA and BHT; and formic acid.

28. (New) Process according to claim 9, by dissolving 0.1-10% of the antioxidant, preferred 0.3-2%, in formic acid, acetic acid and/or propionic acid of concentration 60-100%.

29. (New) Process according to claim 9, for preparation of a silage aid comprising 2,6-di-*tert*-butyl-4-methylphenol (BHT) and 3-*tert*-butyl-4-hydroxyanisole (BHA), and formic acid, by
a) dissolving BHA in the acid, and
b) subsequently, dissolving BHT in the solution obtained in step a).

30. (New) Process according to claim 10, for preparation of a silage aid comprising 2,6-di-*tert*-butyl-4-methylphenol (BHT) and 3-*tert*-butyl-4-hydroxyanisole (BHA), and formic acid, by
a) dissolving BHA in the acid, and
b) subsequently, dissolving BHT in the solution obtained in step a).

REMARKS

The specification has been amended to insert a cross-reference to the International Application.

The claims have been amended to avoid their multiple dependency, as a result of which new claims 14-30 have been added to the application.

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached page is captioned "Version with markings to show changes made."

Respectfully submitted,

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By



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September 10, 2001

THE COMMISSIONER IS AUTHORIZED
TO CHARGE ANY DEFICIENCY IN THE
FEES FOR THIS PAPER TO DEPOSIT
ACCOUNT NO. 23-0975

Version with Markings to
Show Changes Made

Claims

1. Silage aid comprising at least one antioxidant selected from the group
2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA),
5 *tert*-butylhydroquinone (TBHQ), tocopherol and gallates; at least one short chain
carboxylic acid; and optionally at least one salt of said acid.
2. Silage aid according to claim 1, wherein the short chain carboxylic acid is formic acid,
acetic acid and/or propionic acid.
- 10 3. Silage aid according to claim 1 ~~or 2~~, wherein the short chain carboxylic acid is formic
acid of concentration 60-100%, preferred 83-98%.
4. Silage aid according to claim 1 ~~or 2~~ ^{claim 1}, wherein the short chain carboxylic acid is
15 acetic acid or propionic acid of concentration 60-100%, preferred 80-100%.
5. Silage aid according to ~~any of the preceding claims~~ ^{claim 1}, wherein 0.1-10% of the
antioxidant, preferred 0.3-2%, is dissolved in the short chain carboxylic acid.
- 20 6. Silage aid according to ~~any of the preceding claims~~ ^{claim 1}, comprising an antioxidant selected
from the group BHA, TBHQ and propyl galate (PG); and formic acid.
7. Silage aid according to ~~any of the preceding claims~~ ^{claim 1}, comprising BHA and BHT; and
formic acid.
- 25 8. Process for preparation of a silage aid comprising at least one antioxidant selected from
the group 2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA),
tert-butylhydroquinone (TBHQ), tocopherol and gallates; a short chain carboxylic acid;
and optionally at least one salt of said acid, by dissolving the antioxidants in the acid.
- 30 9. Process according to claim 8, by dissolving 0.1-10% of the antioxidant, preferred
0.3-2%, in a short chain carboxylic acid of concentration 60-100%.

10. Process according to claim 8 ~~or 9~~, by dissolving 0.1-10% of the antioxidant, preferred 0.3-2%, in formic acid, acetic acid and/or propionic acid of concentration 60-100%.

11. Process according to claim 8, ~~9 or 10~~, for preparation of a silage aid comprising

5 2,6-di-*tert*-butyl-4-methylphenol (BHT) and 3-*tert*-butyl-4-hydroxyanisole (BHA), and formic acid, by

a) dissolving BHA in the acid, and

b) subsequently, dissolving BHT in the solution obtained in step a).

10 12. Use of a silage aid comprising at least one antioxidant selected from the group 2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA), *tert*-butylhydroquinone (TBHQ), tocopherol and gallates; a short chain carboxylic acid; and optionally at least one salt of said acid, for protection of fish oil during a fish silage process.

15 13. Use of a silage aid comprising at least one antioxidant selected from the group 2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA), *tert*-butylhydroquinone (TBHQ), tocopherol and gallates; a short chain carboxylic acid; and optionally at least one salt of said acid, during preservation of organic by-products.

20

WO 00/53033

A new silage aid, process for preparing this silage aid and use of this silage aid

This invention relates to a new silage aid, process for preparation of this silage aid and use of the silage aid.

When preparing silage from for instance fish waste, the raw material is treated with acid to
5 obtain the optimum pH (3.5-4.5) with regard to enzymatic hydrolysis, and to prevent the growth of bacteria during storage.

During the silage process hydrolysis results in the formation of emulsions made up of an acidic water phase, an oil phase and solids. The degree of phase separation in these systems
10 depends upon the type of raw material used. Oxygen is easily transferred through water, which unfortunately gives rise to oxidative degradation of the oil phase. This lipid oxidation results, of course, in a reduced nutritional value of the oil as an ingredient both for feed and for food. Likewise, it implies a reduced quality of the oleochemicals made from fish silage oil. To overcome these problems it would be a great advantage if an antioxidant
15 well suited for the protection of fish oil could be present during the silage process.

The most favourable way to include an antioxidant would be as a constituent of the silage aid. Generally, it would be necessary to have at least 1% antioxidant in the silage aid. Water soluble antioxidants are easily included in an acidic silage aid. Unfortunately though, these
20 compounds will not be able to protect the oil after separation since they will not be associated with the oil phase. Furthermore, the antioxidants that are food approved in Europe and/or the US at present, are mainly fat soluble compounds, *e.g.* 2,6-di-*tert*-butyl-4-methylphenol(BHT), 3-*tert*-butyl-4-hydroxyanisole(BHA), *tert*-butylhydroquinone(TBHQ), tocopherol and the gallates. All of these food approved
25 antioxidants are also commercially available in formulations with emulsifying agents, but unfortunately the emulsifying agents are in general hydrophobic, and thus these formulations are not well suited to an acidic water medium.

The antioxidant ethoxyquin is an exception with regard to the properties described above.
30 Ethoxyquin is a secondary, cyclic amine. Hence, it is well known that ethoxyquin is soluble in acids at low pH (<3), as this implies protonation of the amino group and thereby formation of a hydrophilic salt. As pH rises to 4-5 however, ethoxyquin will be deprotonated and consequently, will again become fat soluble. Thus, by choosing the proper

concentration of acid it is possible to have ethoxyquin in the form of a hydrophilic salt in the silage aid, while during the silage process the salt is deprotonated, becomes fat soluble and consequently will be able to protect the oil against lipid oxidation. Likewise, other antioxidants with an amino group may be suitable for this use. The preparation of
5 ethoxyquin formate salt, and this salt dissolved in formic acid is disclosed in Norwegian patent application no. 851007.

Unfortunately though, ethoxyquin or other known antioxidants with an amino group, are not food approved. Therefore, when food approval has been required, no silage aid soluble
10 antioxidants have been available to date.

Thus it is a main object of the present invention to provide a new silage aid comprising a food approved antioxidant.

15 This and other objects of the invention is achieved by the attached claims.

The invention will be further explained below.

The antioxidants BHT, BHA, tocopherol, TBHQ and propyl gallate (PG) are practically
20 insoluble in water, and experiments have shown that their solubility in mineral acids (hydrogen chloride, sulphuric acid) is also quite low (see Table 1). Unexpectedly however, we have now found that these highly hydrophobic antioxidants are soluble in the short chain carboxylic acids formic, acetic and propionic acid (see Table 1). Further experiments have shown that when using a silage aid comprising BHA, TBHQ or PG dissolved in 85% formic
25 acid, a superior quality of the fish oil product as compared to the product of the same process using only 85% formic acid (Table 2) is obtained. When the silage aid containing antioxidant is blended with the fish waste raw material, the hydrophobic antioxidant is associated with and protects the oil phase. Thus, we have found a method which is convenient on a technical scale and which leads to superior quality of the products of the
30 silage process.

Generally, the amount of silage aid needed will depend upon the type of fish waste used and the choice of acid. Also, the amount of antioxidant needed, may depend upon the raw

material used or the requirements regarding the stability of the isolated oil. This implies that the required amount of antioxidant dissolved in the acid may vary.

Furthermore, we have shown by experiments that the solubility of BHT in formic acid

5 increases when BHA is present in the acid. This was a surprising result.

The silage aid might further comprise additives like, anti-microbiell compounds (e.g. ethyl benzoate or benzoic acid), anti-fungal compounds, anti-corrosive compounds, chelating compounds (e.g. citric acid), compounds improving the handling properties (e.g. glycerol),

10 and oxygen scavengers.

The present invention also comprises to firstly dissolve the said antioxidants in a short chain carboxylic acid, and subsequently adding a mineral acid in the purpose of decreasing pH.

15 The short chain carboxylic acids according to this invention might be used either alone or in combination with their corresponding salts. Further, the aforementioned acids might be used as a mixture or as a mixture together with any of their salts.

The present invention is documented by experiments performed for a fish silage process.

20 This invention will of course also prevail for other processes where acidic preservation is used. Fish silage shall only be considered as an example. The invention is applicable in acidic preservation of other organic by-products like slaughter waste, poultry waste and food waste, as well.

25 **Table 1.** Solubility (weight %) of antioxidants in different acids.

	BHA	TBHQ	PG	BHT	Toco- pherol
5 M hydrogen chloride ^a	< 0.1%	< 0.1%	< 0.1%	< 0.1%	< 0.1%
4 N sulphuric acid ^a	< 0.1%	< 0.1%	< 0.1%	< 0.1%	< 0.1%
85% formic acid ^b	>5%	>5%	>10%	< 0.25%	< 0.5%
98-100% formic acid ^b	>10%	>10%	>10%	< 0.25% ^c	< 0.5%
100% acetic acid ^b	>10%	>10%	>10%	>10%	>10%
100% propionic acid ^b	>10%	>10%	<10%	>10%	>10%

- a) A mixture of antioxidant(40mg) and mineral acid(40g) was thoroughly shaken at 23°C.
 b) A mixture of the chosen amount of antioxidant(10-400mg) and acid(4g) was thoroughly shaken at 23°C. c) 0.25% BHT is not soluble in formic acid. When 0.5% BHA is added to the formic acid, BHT is soluble.

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Example I: Experimental procedure for a lab-scale fish silage process.

The raw material, Atlantic salmon viscera, was ground in a kitchen grinder, and the resulting minced fish waste was thoroughly blended before it was portioned into separate
 10 batches each containing 500 gram. Silage aid (2% vol./wt.: 10mL) 85% formic acid with or without 0.75% wt./vol. of dissolved antioxidant was added, and the content of each batch was mixed to ensure a homogenous distribution. The batches was stored for eight days in an oven at 35±2°C, before a standard procedure for silage work up was conducted. This included warming the silage at 90±1°C for 1 minute, tempering, and finally separating the
 15 oil after centrifugation. The isolated oils were analysed to determine the POV and *p*-AV. Furthermore, weight gain (at 35±1°C) as a function of time was registered to determine the IP of the oils. The results are presented in Table 2.

20 **Example II:** Experimental procedure as in Example I, but the raw material in this series was whole herring. The results are presented in Table 2.

Table 2. Comparison of the results from analysis of various oil quality parameters^a, determined for fish silage oil produced^b using silage aid with or without dissolved
 25 antioxidant.

Example no.	Silage aid ^c	POV	<i>p</i> -AV	IP
I	Control: 85% HCOOH (formic acid)	3	24	1
	85% HCOOH incl. BHA (150ppm)	2	22	19
	85% HCOOH incl. TBHQ (150ppm)	1	9	62
II	Control: 85% HCOOH	29	29	0
	85% HCOOH incl. PG (150ppm)	7	19	4

a) POV(Peroxide value, Ph. Eur. V.3.4.5); determines the amount of primary oxidation
 30 products, hydroperoxides, in the oil. *p*-AV(*p*-Anisidine value, IUPAC 2.504); determines

the amount of some secondary oxidation products, alkenals, in the oil. IP(induction period); a measure of the shelf life of the oil, given as the number of days it takes before the oil shows detectable weight gain due to oxygen absorption. b)The experimental procedure for lab-scale silage experiments is given above in example I. c)The concentration of

5 antioxidant given in parenthesis, is based on the amount of fish waste used in these experiments.

Claims

1. Silage aid comprising at least one antioxidant selected from the group
2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA),
5 *tert*-butylhydroquinone (TBHQ), tocopherol and gallates; at least one short chain
carboxylic acid; and optionally at least one salt of said acid.
2. Silage aid according to claim 1, wherein the short chain carboxylic acid is formic acid,
acetic acid and/or propionic acid.
- 10 3. Silage aid according to claim 1 or 2, wherein the short chain carboxylic acid is formic
acid of concentration 60-100%, preferred 83-98%.
4. Silage aid according to claim 1 or 2 claims, wherein the short chain carboxylic acid is
15 acetic acid or propionic acid of concentration 60-100%, preferred 80-100%.
5. Silage aid according to any of the proceeding claims, wherein 0.1-10% of the
antioxidant, preferred 0.3-2%, is dissolved in the short chain carboxylic acid.
- 20 6. Silage aid according to any of the proceeding claims, comprising an antioxidant selected
from the group BHA, TBHQ and propyl galate (PG); and formic acid.
7. Silage aid according to any of the proceeding claims, comprising BHA and BHT; and
formic acid.
- 25 8. Process for preparation of a silage aid comprising at least one antioxidant selected from
the group 2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA),
tert-butylhydroquinone (TBHQ), tocopherol and gallates; a short chain carboxylic acid;
and optionally at least one salt of said acid, by dissolving the antioxidants in the acid.
- 30 9. Process according to claim 8, by dissolving 0.1-10% of the antioxidant, preferred
0.3-2%, in a short chain carboxylic acid of concentration 60-100%.

10. Process according to claim 8 or 9, by dissolving 0.1-10% of the antioxidant, preferred 0.3-2%, in formic acid, acetic acid and/or propionic acid of concentration 60-100%.

11. Process according to claim 8, 9 or 10, for preparation of a silage aid comprising

5 2,6-di-*tert*-butyl-4-methylphenol (BHT) and 3-*tert*-butyl-4-hydroxyanisole (BHA), and formic acid, by

a) dissolving BHA in the acid, and

b) subsequently, dissolving BHT in the solution obtained in step a).

10 12. Use of a silage aid comprising at least one antioxidant selected from the group

2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA), *tert*-butylhydroquinone (TBHQ), tocopherol and gallates; a short chain carboxylic acid; and optionally at least one salt of said acid, for protection of fish oil during a fish silage process.

15

13. Use of a silage aid comprising at least one antioxidant selected from the group

2,6-di-*tert*-butyl-4-methylphenol (BHT), 3-*tert*-butyl-4-hydroxyanisole (BHA), *tert*-butylhydroquinone (TBHQ), tocopherol and gallates; a short chain carboxylic acid; and optionally at least one salt of said acid, during preservation of organic by-products.

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#6

DECLARATION AND POWER OF ATTORNEY FOR U.S. PATENT APPLICATION

☐ Original ☐ Supplemental ☐ Substitute ☒ PCT ☐ DESIGN

As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that I verily believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Title: A NEW SILAGE AID, PROCESS FOR PREPARING THIS SILAGE AID AND USE OF THIS SILAGE AID

of which is described and claimed in:

- ☐ the attached specification, or
☐ the specification in application Serial No. , filed , and with amendments through , or
☒ the specification in International Application No. PCT/NO00/00079, filed March 6, 2000, and as amended on (if applicable).

I hereby state that I have reviewed and understand the content of the above-identified specification, including the claims, as amended by any amendment(s) referred to above.

I acknowledge my duty to disclose to the Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim priority benefits under Title 35, United States Code, §119 (and §172 if this application is for a Design) of any application(s) for patent or inventor's certificate listed below and have also identified below any application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

COUNTRY	APPLICATION NO.	DATE OF FILING	PRIORITY CLAIMED
Norway	19991111	March 8, 1999	yes

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NO.	U.S. FILING DATE	STATUS: PATENTED, PENDING, ABANDONED

And I hereby appoint Michael R. Davis, Reg. No. 25,134; Matthew M. Jacob, Reg. No. 25,154; Warren M. Cheek, Jr., Reg. No. 33,367; Nils Pedersen, Reg. No. 33,145; Charles R. Watts, Reg. No. 33,142; and Michael S. Huppert, Reg. No. 40,268, who together constitute the firm of WENDEROTH, LIND & PONACK, L.L.P., as well as any other attorneys and agents associated with Customer No. 000513, to prosecute this application and to transact all business in the U.S. Patent and Trademark Office connected therewith.

I hereby authorize the U.S. attorneys and agents named herein to accept and follow instructions from NORSK HYDRO ASA as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. attorneys and myself. In the event of a change in the persons from whom instructions may be taken, the U.S. attorneys named herein will be so notified by me.

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I further declare that all statements made herein of my own knowledge are true, and that all statements on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

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The above application may be more particularly identified as follows:

U.S. Application Serial No. 09/936,165 Filing Date September 10, 2001

Applicant Reference Number P99023 NWe:ELS Atty Docket No. 2001 1245A

Title of Invention A NEW SILAGE AID, PROCESS FOR PREPARING THIS SILAGE AID AND USE OF THIS SILAGE AID